PATENT SPECIFICATION (11) 1 438 205

1 438 205

(21) Application No. 37419/74 (22) Filed 27 Aug. 1974
(44) Complete Specification published 3 June 1976
(51) INT CL² A61K 7/16
(52) Index at acceptance
A5B 301 30Y 385 38Y 39X 406 40Y 410 41Y 772

(72) Inventors SHIGEO TSUCHIYA, SHIGEYOSHI HATASA and ISAO IIOKA

(19)

(54) DENTIFRICE COMPOSITION

(71) We, THE LION DENTIFRICE CO. LTD., of 3—7, Honjo 1-chome, Sumida-ku, Tokyo, Japan, a Japanese Company, do hereby declare the invention, for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a composition for oral use such as a dentifrice.

It is known to use flavours of various types blended with medicinal ingredients in compositions for oral use, to provide a refreshing feeling. However conventional refreshing agents impart relatively weak and transitive effects. Menthol has been most commonly used as a flavouring agent, however a relatively large amount of menthol is required for providing a satisfactory refreshing sensation. The addition of a too large an amount of menthol causes a bitter taste. Accordingly, the amount of menthol should be limited. When propyleneglycol is used for forming a dentifrice composition together with menthol a severe bitter taste is given. Sodium α-olefin sulfonates or sodium alkylsulfates are used for forming a dentifrice composition together with menthol but a severe bitter taste can remain and orange juice tastes bitter after use of the dentifrice.

It is an object of this invention to provide dentifrice composition which impart a pleasing and long lasting refreshing sensation in the mouth. It is another object of this invention to provide dentifrice composition which promotes salivation and a refreshing feeling, and provides analgesic activity caused by local anethetic effect whereby the oral administration such as a tooth brushing can be comfortable, even when the user suffers from toothache.

According to the invention there is provided a composition for oral use such as a dentifrice, mouthwash or gargle which includes N-isobutyl-2,6,8-decatrienamide in a suitable base or carrier.

The active ingredient can be an essential oil containing N-isobutyl-2,6,8-decatrienamides such as the essential oils derived from the plants Spilanthes Acmella Linne var oleaceae Clarke, Spilanthes oleracea Jacquin containing spilanthol; Erigeron Affinis D.C. containing affinine.

Synthesized compounds can be used to form the active ingredient. The active ingredients N-isobutyl-2,6,8-decatrienamide can be prepared from the above-mentioned plants, by extracting the dry grass or the flower heads, which are rich in the active ingredient, with ether, then separating ether from the extracts by distillation and removing volatile components from the oleoresin residue by steam distillation.

The residue can be extracted with ethylalcohol and the insoluble impurities removed. The alcohol can then be stripped off and the product extracted with ether and then the ether stripped off and the residue saponified with 10% alcoholic potassium hydroxide so as to decompose the oil and fat impurities.

The resulting alcoholic solution can then be diluted with a large amount of water and finally extracted with ether to obtain the active ingredient. The N-isobutyl-2,6,8-decatrienamide or essential oil containing the same can be incorporated in a desirable base to form a composition for oral use such as a dentifrice, mouth-wash or gargle.

It is preferable to combine N-isobutyl-2,6,8-decatrienamide with another flavouring agent especially menthol, in an amount of 0.01—10.0% by weight especially 0.1—5.0% by weight of the total flavouring agent. It is especially preferable to combine 1 part by weight of N-isobutyl-2,6,8-decatrienamide with 1—1000 parts especially 50—500 parts by weight of menthol.

Peppermint essential oil can be used to provide menthol. Other flavours such

as anethol, carvone, methyl salicylate (oil of wintergreen) can also be effectively blended in.

In a dentifrice it is preferable to incorporate 0.001—5.00% by weight especially 0.005—1.0% by weight, based on the total weight, of N-isobutyl-2,6,8-decatrienamide to the base.

The dentifrice compositions according to this invention can be prepared in the form e.g. of dental cream, tooth cake or tooth powder.

The abrasive ingredients contained in the dentifrice composition can be any of the types conventionally used. Typical abrasive ingredients include insoluble sodium metaphosphate, tricalcium phosphate, calcium hydrogen phosphate dihydrate, anhydrous calcium hydrogen phosphate, calcium pyrophosphate, magnesium orthophosphate, trimagnesium phosphate, calcium carbonate, alumina, silica and mixture thereof.

In addition, a sweetening agent such as saccharin, other flavouring agents, a preservative ingredient such as sodium benzoate, a coloring agent, a binder or other ingredients for dentifrice compositions can also be blended in.

The N-isobutyl-2,6,8-decatrienamide can be combined with menthol and propyleneglycol and sodium olefinsulfonate as base ingredients.

Sodium alkylsulfates such as sodium laurylsulfate can replace the sodium olefinsulfonate.

Even though menthol is combined with propyleneglycol and a sodium olefinsulfonate or sodium alkylsulfate, the bitter taste can be prevented by including N-isobutyl-2,6,8-decatrienamide.

Especially, a taste of beverage or food after cleaning with a dentifrice, can be improved by combining N-isobutyl-2,6,8-decatrienamide with menthol and propyleneglycol, a sodium olefinsulfonate or sodium laurylsulfate.

The preferred sodium olefinsulfonate has the formula

$$R—CH=CH—(CH_2)_y—SO_3Na$$

wherein R represents C_{7-21} alkyl group y represents 0 or an integer of 1—10.

Typical sodium olefinsulfonates include sodium tetradecenesulfonate, hexadecenesulfonate, dodecenesulfonate and octadecenesulfonate.

It is especially preferable to combine N-isobutyl-2,6,8-decatrienamide with gambir, ginger or zanthoxylum together with menthol to remove a metallic taste.

The sodium olefinsulfonate or alkylsulfate is preferably within the range 0.1—5 wt% of the total.

Menthol is preferably within the range 0.01—5 wt% of the total.

Gambir, ginger or zanthoxylum is preferably within the range 0.01—5 wt% of the total.

Propyleneglycol is preferably within the range 1—10 wt% of the total.

From the economic viewpoint, it is preferable to incorporate purified essential oils of Spilanthes Acmella Linne var oleraceae Clarke and Spilanthes oleracea Jacquin containing spilanthol; Erigeron Affinis D.C. containing Afinine.

In order to illustrate the invention, the results of incorporation of spilanthol especially, combination of spilanthol and menthol are stated.

The compositions shown in the Table 1 were prepared by a conventional process for preparing tooth paste.

TABLE 1

Ingredient	Composition (by weight)					
	1	2	3	4	5	6
Calcium hydrogen phosphate	50%	50%	50%	50%	50%	50%
Na carboxymethyl cellulose	1.0	1.0	1.0	1.0	1.0	1.0
Na dodecenesulfonate	2.0	2.0	2.0	2.0	—	—
Na laurylsulfate	—	—	—	—	2.0	2.0
Glycerin	20.0	20.0	20.0	20.0	20.0	20.0
Propyleneglycol	5.0	5.0	5.0	5.0	5.0	5.0
Saccharin	0.2	0.2	0.2	0.2	0.2	0.2
Menthol	1.0	1.0	1.0	1.0	1.0	1.0
Gambir	—	0.2	—	—	—	—
Ginger	0.2	—	—	—	0.2	0.2
Zanthoxylum	—	—	0.2	—	—	—
N-isobutyl-2,6,8-decatrienamide	0.01	0.01	0.01	0.01	0.01	0.01
Water	q.v.	q.v.	q.v.	q.v.	q.v.	q.v.
Total	100.0	100.0	100.0	100.0	100.0	100.0

The compositions 1—6 were tested by 20 panel members who were well trained.

The panel tests were conducted by a conventional sense testing method.

All of the panel members felt long lasting time refreshing feeling and no bitter taste. When N-isobutyl decatrienamide is removed from the dentifrice composition, all of the panel members felt a bitter taste.

In accordance with the compositions for oral use of this invention wherein N-isobutyl-2,6,8-decatrienamides in purified essential oils of Spilanthes Acmella Linne var oleraceae Clarke and Spilanthes oleracea Jacquin, etc., are incorporated in a desirable base of the compositions for oral use, the following advantages are found.

(1) The refreshing feeling can be remarkably increased.

(2) The duration of the refreshing feeling can be remarkably prolonged.

(3) When a combination of spilanthol and menthol is employed, the characteristics of menthol can be enhanced.

(4) A local anesthetic property can be provided without providing an astringent taste during tooth brushing, thereby allowing comfortable and effective mouth washing and tooth brushing even when the user suffers from toothache. Accordingly, it can be used for a medication by a dentist.

(5) The refreshing sensation is not accompanied by any bitter taste.

(6) Salivation is promoted so that the appetite is improved.

(7) No bitter taste from the combination of menthol and propyleneglycol is found.

(8) No bitter taste from the combination of menthol and sodium olefin-sulfonate is found.

(9) No metallic taste is felt when gambir, ginger or zanthoxylum are combined.

Certain specific examples of this invention are hereafter given for purposes of illustration only and are not intended to be limiting. All parts and percents are shown by weight.

Flavouring composition (1):

Formulation:			
	Menthol	40	parts
	N-isobutyl-2,6,8-decatrienamide	0.5	,,
	Anethole	10	,,
	Carvon	20	,,
	Methyl salicylate	20	,,
	Ginger	10	,,

Flavouring composition (2):

Formulation			
	Menthol	30	parts
	N-isobutyl-2,6,8-decatrienamide	0.3	,,
	Anethole	15	,,
	Carvon	20	,,
	Methyl salicylate	30	,,
	Zanthoxylum	2	,,

Example 1.

Tooth-paste:

Calcium hydrogen phosphate	50.0%	50.0%
Na—C.M.C.	1.0	1.0
Na dodecensulfonate	1.5	1.5
Glycerin	20.0	20.0
Propyleneglycol	5.0	5.0
Saccharin	0.1	0.1
Flavouring Composition (1)	2.0	—
Flavouring Composition (2)	—	2.0
Water	q.v	q.v.
	ad. 100.0	ad. 100.0

Example 2.

Tooth-paste:

Insoluble Sodium metaphosphate	30.0%	30.0%
Calcium hydrogen phosphate	20.0	20.0
Irish moss	1.3	1.3
Liquid sorbitol	30.0	30.0
Propyleneglycol	5.0	5.0
Na octadecensulfonate	2.0	2.0
Saccharin	0.1	0.1
Flavouring Composition (1)	2.0	—
Flavouring Composition (2)	—	2.0
Water	q.v.	q.v.
	ad. 100.0	ad. 100.0

Example 3.

Tooth-paste:

Calcium carbonate	45.0%	45.0%
Na—C.M.C.	1.2	1.2
Liquid sorbitol	25.0	25.0
Propyleneglycol	5.0	5.0
Na-hexadecensulfonate	1.5	1.5
Saccharin	0.15	0.15
Flavouring Composition (1)	1.4	—
Flavouring Composition (2)	—	1.4
Water	q.v.	q.v.
	ad. 100.0	ad. 100.0

The tooth paste of the Examples 1—3, respectively have a good taste and give the described refreshing sensation and the mild local anesthetic property.

WHAT WE CLAIM IS:—

1. A composition for oral use such as a dentifrice, mouthwash or gargle which includes N-isobutyl-2,6,8-decatrienamide in a suitable base or carrier.

2. A composition as claimed in claim 1 which includes menthol.

3. A composition as claimed in claim 1 or claim 2 which includes propyleneglycol.

4. A composition as claimed in any preceding claim which includes a sodium olefinsulfonate.

5. A composition as claimed in any preceding claim which includes a sodium alkylsulfate.

6. A composition as claimed in any preceding claim wherein the N-isobutyl-2,6,8-decatrienamide is contained in at least one of the essential oils from Spilanthes Acmella Linne var oleraceae Clarke, Spilanthes oleracea Jacquin and Erigeron Affinis D.C.

7. A composition as claimed in any preceding claim which includes 0.001—5 wt.% of total weight of gambir, ginger and/or zanthoxylum.

8. A composition as claimed in any preceding claim which comprises a base, 0.1—5 wt.% of sodium olefinsulfonate, 0.001—5.0 wt.% of N-isobutyl-2,6,8-decatrienamide and 0.01—5 wt.% of menthol.

9. A composition as claimed in any preceding claim which comprises a base, 0.1—5 wt.% of sodium laurylsulfate, 0.001—5 wt.% of N-isobutyl-2,6,8-decatrienamide and 0.01—5 wt.% of menthol.

10. A composition as claimed in any preceding claim in the form of a toothpaste.

11. A toothpaste substantially as described herein with reference to any one of the Examples.

R. G. C. JENKINS & CO.,
Chartered Patent Agents,
Chancery House,
53/64 Chancery Lane,
London WC2A 1QU.
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1976.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.